

09/914029

Practitioner's Docket No. U013610-2

Optional Customer No. Bar Code



00140

PATENT TRADEMARK OFFICE

CHAPTER II

**TRANSMITTAL LETTER
TO THE UNITED STATES ELECTED OFFICE (EO/US)**

(ENTRY INTO U.S. NATIONAL PHASE UNDER CHAPTER II)

PCT/ES99/00060	10 MARCH 1999
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED
PIGMENTS BASED ON SILICA AND IRON OXIDE AND PROCESS FOR THE MANUFACTURE THEREOF	
TITLE OF INVENTION	

1. VICENTE BAGAN VARGAS
2. ANTONIO BLASCO FUENTES
3. FRANCISCO NEGRE MEDALL
4. VICENTE BELTRAN PORCAR
5. BENITO CERVANTES RODRIGUEZ

APPLICANT(S)

Box PCT
Assistant Commissioner for Patents
Washington D.C. 20231
ATTENTION: EO/US

CERTIFICATION UNDER 37 C.F.R. 1.10*
(Express Mail label number is mandatory.)
(Express Mail certification is optional.)

I hereby certify that this correspondence and the documents referred to as attached therein are being deposited with the United States Postal Service on this date August 22, 2001, in an envelope as "Express Mail Post Office to Addressee," Mailing Label Number EL728214728US, addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

IBIS CARRILLO*(type or print name of person mailing paper)*

Signature of person mailing paper

WARNING: Certificate of mailing (first class) or facsimile transmission procedures of 37 C.F.R. 1.8 cannot be used to obtain a date of mailing or transmission for this correspondence.

***WARNING:** Each paper or fee filed by "Express Mail" must have the number of the "Express Mail" mailing label placed thereon prior to mailing. 37 C.F.R. 1.10(b).
 "Since the filing of correspondence under § 1.10 without the Express Mail mailing label thereon is an oversight that can be avoided by the exercise of reasonable care, requests for waiver of this requirement will **not** be granted on petition." Notice of Oct. 24, 1996, 60 Fed. Reg. 56,439, at 56,442.

NOTE: The completion of those filing requirements that can be made at a time later than 30 months from the priority date results from the Commissioner exercising his judgment under the authority granted under 35 USC 371(d). The filing receipt will show the actual date of receipt of the last item completing the entry into the national phase. See 37 C.F.R. §1.491 which states: "An international application enters the national state when the applicant has filed the documents and fees required by 35 USC 371(c) within the periods set forth in § 1.494 and § 1.495."

WARNING: Where the items are those which can be submitted to complete the entry of the international application into the national phase are subsequent to 30 months from the priority date the application is still considered to be in the international state and if mailing procedures are utilized to obtain a date the express mail procedure of 37 C.F.R. §1.10 must be used (since international application papers are not covered by an ordinary certificate of mailing - See 37 C.F.R. §1.8.

NOTE: Documents and fees must be clearly identified as a submission to enter the national state under 35 USC 371 otherwise the submission will be considered as being made under 35 USC 111. 37 C.F.R. § 1.494(f).

1. Applicant herewith submits to the United States Elected Office (EO/US) the following items under 35 U.S.C. 371:

- a. ☒ This express request to immediately begin national examination procedures (35 U.S.C. 371(f)).
- b. ☒ The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees (37 C.F.R. § 1.492) as indicated below:

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JC03 Rec'd PCT/TC 22 AUG 2001

2.Fees

CLAIMS FEE	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
[]*	TOTAL CLAIMS	19 - 20 =		x \$ 18.00 =	\$
	INDEPENDENT CLAIMS	2 - 3 =		x \$ 80.00 =	
	MULTIPLE DEPENDENT CLAIM(S) (if applicable) + \$270.00				
BASIC FEE**	<p>[] U.S. PTO WAS INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY Where an International preliminary examination fee as set forth in § 1.482 has been paid on the international application to the U.S. PTO:</p> <p>[] and the international preliminary examination report states that the criteria of novelty, inventive step (non-obviousness) and industrial activity, as defined in PCT Article 33(2) to (4) have been satisfied for all the claims presented in the application entering the national stage (37 CFR 1.492(a)(4)) \$100.00</p> <p>[] and the above requirements are not met (37 CFR 1.492(a)(1)) \$690.00</p> <p>[x] U.S. PTO WAS NOT INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY Where no international preliminary examination fee as set forth in § 1.482 has been paid to the U.S. PTO, and payment of an international search fee as set forth in § 1.445(a)(2) to the U.S. PTO:</p> <p>[] has been paid (37 CFR 1.492(a)(2)) \$710.00</p> <p>[x] has not been paid (37 CFR 1.492(a)(3)) \$1,000.00</p> <p>[] where a search report on the international application has been prepared by the European Patent Office or the Japanese Patent Office (37 CFR 1.492(a)(5)) \$860.00</p>				
	Total of above Calculations				1000.00
SMALL ENTITY	Reduction by ½ for filing by small entity, if applicable. Statement may also be filed. (note 37 CFR 1.9, 1.27, 1.28)				-
	Subtotal				
	Total National Fee				\$1000.00
	Fee for recording the enclosed assignment document \$40.00 (37 CFR 1.21(h)). (See Item 13 below). See attached "ASSIGNMENT COVER SHEET".				
TOTAL	Total Fees enclosed				\$1000.00

*See attached Preliminary Amendment Reducing the Number of Claims.

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- (Transmittal Letter to the United States Elected Office (EO/US)—page 4 of 8) 13-18

10. ☒ An oath or declaration of the inventor (35 U.S.C. 371(c)(4)) complying with 35 U.S.C. 115
- a. ☐ was previously submitted by applicant on _____
Date
- b. ☐ is submitted herewith, and such oath or declaration
- i. ☐ is attached to the application.
- ii. ☐ identifies the application and any amendments under PCT Article 19 that were transmitted as stated in points 3(b) or 3(c) and 5(b); and states that they were reviewed by the inventor as required by 37 C.F.R. 1.70.
- c. ☒ will follow.

Other document(s) or information included:

11. ☒ An International Search Report (PCT/ISA/210) or Declaration under PCT Article 17(2)(a):
- a. ☐ is transmitted herewith.
- b. ☒ has been transmitted by the International Bureau.
Date of mailing (from form PCT/IB/308): 14 SEPTEMBER 2000.
- c. ☐ is not required, as the application was searched by the United States International Searching Authority.
- d. ☐ will be transmitted promptly upon request.
- e. ☐ has been submitted by applicant on _____
Date
12. ☒ An Information Disclosure Statement under 37 C.F.R. 1.97 and 1.98:
- a. ☐ is transmitted herewith.
Also transmitted herewith is/are:
- ☐ Form PTO-1449 (PTO/SB/08A and 08B).
- ☐ Copies of citations listed.
- b. ☒ will be transmitted within THREE MONTHS of the date of submission of requirements under 35 U.S.C. 371(c).
- c. ☐ was previously submitted by applicant on _____
Date
13. ☐ An assignment document is transmitted herewith for recording.

A separate ☐ "COVER SHEET FOR ASSIGNMENT (DOCUMENT) ACCOMPANYING NEW PATENT APPLICATION" or ☐ FORM PTO 1595 is also attached.

14. ☒ Additional documents:
- a. ☒ Copy of request (PCT/RO/101)
 - b. ☒ International Publication No. WO 00/53680
 - i. ☒ Specification, claims and drawing
 - ii. ☐ Front page only
 - c. ☒ Preliminary amendment (37 C.F.R. § 1.121)
 - d. ☒ Other

FORM PCT/IPEA/401: REPLY TO THE WRITTEN RESPONSE

FORM PCT/IPEA/408: FORM PCT/IPEA/416

15. ☐ The above checked items are being transmitted
- a. ☐ before 30 months from any claimed priority date.
 - b. ☐ after 30 months.
16. ☐ Certain requirements under 35 U.S.C. 371 were previously submitted by the applicant on _____, namely:
- _____
- _____
- _____

AUTHORIZATION TO CHARGE ADDITIONAL FEES

WARNING: *Accurately count claims, especially multiple dependent claims, to avoid unexpected high charges if extra claims are authorized.*

NOTE: *"A written request may be submitted in an application that is an authorization to treat any concurrent or future reply, requiring a petition for an extension of time under this paragraph for its timely submission, as incorporating a petition for extension of time for the appropriate length of time. An authorization to charge all required fees, fees under § 1.17, or all required extension of time fees will be treated as a constructive petition for an extension of time in any concurrent or future reply requiring a petition for an extension of time under this paragraph for its timely submission. Submission of the fee set forth in § 1.17(a) will also be treated as a constructive petition for an extension of time in any concurrent reply requiring a petition for an extension of time under this paragraph for its timely submission." 37 C.F.R. § 1.136(a)(3).*

NOTE: *"Amounts of twenty-five dollars or less will not be returned unless specifically requested within a reasonable time, nor will the payer be notified of such amounts; amounts over twenty-five dollars may be returned by check or, if requested, by credit to a deposit account." 37 C.F.R. § 1.26(a).*

☒ The Commissioner is hereby authorized to charge the following additional fees that may be required by this paper and during the entire pendency of this application to Account No. 12-0425.

☒ 37 C.F.R. 1.492(a)(1), (2), (3), and (4) (filing fees)

WARNING: *Because failure to pay the national fee within 30 months without extension (37 C.F.R. § 1.495(b)(2)) results in abandonment of the application, it would be best to always check the above box.*

☐ 37 C.F.R. 1.492(b), (c) and (d) (presentation of extra claims)

NOTE: *Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must*

only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 C.F.R. § 1.492(d)), it might be best not to authorize the PTO to charge additional claim fees, except possible when dealing with amendments after final action.

- ☒ 37 C.F.R. 1.17 (application processing fees)
- ☒ 37 C.F.R. 1.17(a)(1)-(5)(extension fees pursuant to § 1.136(a).
- ☒ 37 C.F.R. 1.18 (issue fee at or before mailing of Notice of Allowance, pursuant to 37 C.F.R. 1.311(b))

NOTE: Where an authorization to charge the issue fee to a deposit account has been filed before the mailing of a Notice of Allowance, the issue fee will be automatically charged to the deposit account at the time of mailing the notice of allowance. 37 C.F.R. § 1.311(b).

NOTE: 37 C.F.R. 1.28(b) requires "Notification of any change in loss of entitlement to small entity status must be filed in the application . . . prior to paying, or at the time of paying . . . issue fee." From the wording of 37 C.F.R. § 1.28(b): (a) notification of change of status must be made even if the fee is paid as "other than a small entity" and (b) no notification is required if the change is to another small entity.

- ☐ 37 C.F.R. § 1.492(e) and (f) (surcharge fees for filing the declaration and/or filing an English translation of an International Application later than 30 months after the priority date).


SIGNATURE OF PRACTITIONER

WILLIAM R. EVANS
(type or print name of practitioner)

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Customer No.: 00140

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New York, N.Y. 10023

2022220 62047660

09/914029

JC03 Rec'd POT/TO 22 AUG 2001

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: VICENTE BAGAN VARGAS, ET AL.

Filed: AUGUST 22, 2001

For: PIGMENTS BASED ON SILICA AND IRON OXIDE AND PROCESS FOR THE
MANUFACTURING THEREOF

Attorney Docket No.: U013610-2

Assistant Commissioner for Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Please cancel claim 14

Please amend the above application as follows.

IN THE CLAIMS:

Claim 5 (amended) A process according to claim 3, in which the blend from stage a) is carried out by milling.

CERTIFICATION UNDER 37 C.F.R. 1.10*

(Express Mail label number is **mandatory**.)

(Express Mail certification is optional.)

I hereby certify that this correspondence and the documents referred to as attached therein are being deposited with the United States Postal Service on this date August 21, 2001, in an envelope as "Express Mail Post Office to Addressee," Mailing Label Number EL728214728US, addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

IBIS CARRILLO

(type or print name of person mailing paper)



Signature of person mailing paper

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Claim 6 (amended) A process according to claim 4, in which the mixture from stage a) is carried out by dispersion, preferably in water

Claim 7 (amended) A process according to claim 5, in which the agglomeration of stage b) consists of a granulation.

Claim 8 (amended) A process according to claim 6, in which the agglomeration of stage b) consists of drying by atomisation.

Claim 9 (amended) A process according to claim 2, in which, before stage c) there is a prior pre-calcination step.

Claim 10 (amended) A process according to claim 2, in which, after stage c), there is a cooling step, prior to blending 10 of the resulting pigment.

Claim 11 (amended) A process according to claim 2, in which step e) preferably consists of grinding or milling.

Claim 12 (amended) Pigments obtained according to the process of claim 2.

Claim 15 (amended) Use according to claim 13, characterised in that the source of silica is obtained from condensation of gases evolved during the manufacture of silicon metal and/or alloys thereof.

Claim 16 (amended) Use of the pigments of claim 1, alone or in blends with other materials, as integrants in the compositions of enamels, glasses, ceramics, cements, plastics, laminates, graphic inks and rubber.

Claim 17 (amended) Use of the pigments of claim 1, alone or in blends with other materials, in the surface decoration of enamels, glasses, ceramics, cements, plastics, laminates, graphic inks and rubber.

Claim 18 (amended) A ceramic product characterised in that it includes in its composition the pigments of claim 1.

Table 1. Demographic characteristics of the study population	
Characteristic	Frequency (%)
Age (years)	
< 18	10 (10.0)
18-24	25 (25.0)
25-34	30 (30.0)
35-44	20 (20.0)
45-54	15 (15.0)
55-64	10 (10.0)
65-74	5 (5.0)
≥ 75	5 (5.0)
Gender	
Male	45 (45.0)
Female	55 (55.0)
Ethnicity	
White	30 (30.0)
Black	20 (20.0)
Hispanic	15 (15.0)
Asian	10 (10.0)
Other	25 (25.0)
Marital status	
Married	35 (35.0)
Single	20 (20.0)
Divorced	15 (15.0)
Widowed	10 (10.0)
Never married	5 (5.0)
Education level	
High school or less	20 (20.0)
Some college	15 (15.0)
College graduate	10 (10.0)
Postgraduate	5 (5.0)
Unknown	50 (50.0)
Annual income (\$)	
< 10,000	10 (10.0)
10,000-19,999	15 (15.0)
20,000-29,999	10 (10.0)
30,000-39,999	5 (5.0)
40,000-49,999	5 (5.0)
50,000-59,999	5 (5.0)
60,000-69,999	5 (5.0)
70,000-79,999	5 (5.0)
80,000-89,999	5 (5.0)
90,000-99,999	5 (5.0)
≥ 100,000	5 (5.0)
Unknown	50 (50.0)

Respectfully submitted,

WILLIAM R. EVANS
LADAS & PARRY
26 WEST 61ST STREET
NEW YORK, NY 10023
REG. NO: 25,858 (212) 708-1930

MARKED UP COPY

5. A process according to claim[s 2 and] 3, in which the blend from stage a) is carried out by milling

6. A process according to claim[s 2 and] 4, in which the mixture from stage a) is carried out by dispersion, preferably in water.

7. A process according to claim[s 2, 3 and] 5, in which the agglomeration of stage b) consists of a granulation.

8. A process according to claim[s 2, 4 and] 6, in which the agglomeration of stage b) consists of drying by atomisation.

9. A process according to claim[s] 2 [to 8], in which, before stage c) there is a prior pre-calcination step.

10. A process according to claim[s] 2 [to 9], in which, after stage c), there is a cooling step, prior to blending 10 of the resulting pigment.

11. A process according to claim[s] 2 [to 10], in which step e) preferably consists of grinding or milling,

12. Pigments obtained according to the process of claim[s] 2 [to 11].

15. Use according to claim[s] 13 [and 14], characterised in 20 that the source of silica is obtained from condensation of gases evolved during the manufacture of silicon metal and/or alloys thereof.

16. Use of the pigments of claim[s] 1 [or 12], alone or in blends with other materials, as integrants in the compositions of enamels, glasses, ceramics, cements, plastics, laminates, graphic inks and rubber.

17. Use of the pigments of claim[s] 1 [or 12], alone or in blends with other materials, in the surface decoration of enamels, glasses, ceramics, cements, plastics, Laminates, graphic inks and rubber.

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18. A ceramic product characterised in that it includes in its composition the pigments of claim[s] 1 [or 12].

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Practitioner's Docket No. U 013610-2

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

☒ In re application of: **Vicente BAGAN VARGAS, ET AL.**

Application No.: **09/914,029**

Group No.:

Filed: **August 22, 2001**

Examiner:

For: **Pigments Based on Silica and Iron Oxide and Process for the Manufacture Thereof**

☐ *Patent No.:

Issue Date:

*NOTE: Insert name(s) of inventor(s) and title also for patent Where statement is with respect to a maintenance fee payment, also insert application number and filing date, and add Box M. Fee to address.

STATEMENT CLAIMING SMALL ENTITY STATUS (37 CFR 1.9(c-f) and 1.27(b-d))

With respect to the invention described in

☐ the specification filed herewith.

☒ application no. 09/914,029, filed 22 August 2001

☐ patent no. _____ issued _____.

I. IDENTIFICATION AND RIGHTS AS A SMALL ENTITY

I hereby state that I am

(complete either (a), (b), (c) or (d) below)

(a) Independent Inventor

☐ a below named independent inventor, and that I qualify as an independent inventor, as defined in 37 CFR 1.9(c), for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code, to the Patent and Trademark Office.

(b) Noninventor Supporting a Claim by Another

☐ making this statement to support a claim by

for a small entity status for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code. I hereby state that I would qualify as an independent inventor as defined in 37 CFR 1.9(c) for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code, if I had made the above identified invention.

(c) Small Business Concern

☐ the owner of the small business concern identified below:

check
one →

☒ an official of the small business concern empowered to act on behalf of the concern identified below:

ITACA, INNOVACIONES TECNICAS APLICADAS A CERAMICAS
Name of Concern AVANZADAS, S.A.
Address of Concern Partida Rambleta s/n, 12191 Puebla Ternes, Castellón
Spain and

that the above identified small business concern qualifies as a small business concern, as defined in 13 CFR 121.3-18, and reproduced in 37 CFR 1.9(d), for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons. For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has the power to control both.

(d) Non-Profit Organization

☐ an official empowered to act on behalf of the nonprofit organization identified below:

Name of Organization _____
Address of Organization _____

TYPE OF ORGANIZATION

- ☐ University or Other Institution of Higher Education
☐ Tax Exempt Under Internal Revenue Service Code (26 USC 501(a) and 501(c) (3))
☐ Nonprofit Scientific or Educational Under Statute of State of the United States of America
(Name of State _____)
(Citation of Statute _____)
☐ Would Qualify as Tax Exempt Under Internal Revenue Service Code (26 USC 501(a) and 501(c) (3)), if Located in the United States of America
☐ Would Qualify as Nonprofit Scientific or Educational Under Statute of State of the United States of America, if Located in the United States of America
(Name of State _____)
(Citation of Statute _____)

and that the nonprofit organization identified above qualifies as a nonprofit organization, as defined in 37 CFR 1.9(e), for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code.

II. OWNERSHIP OF INVENTION BY DECLARANT

I hereby state that rights under contract or law remain with and/or have been conveyed to the above identified

☐ person
(item (a) or (b) above)

☐ concern
(item (c) above)

☐ organization
(item (d) above)

EXCEPT, that if the rights held are not exclusive, each individual, concern or organization having rights to the invention is listed below* and no rights to the invention are held (1) by any person who could not be classified as an independent inventor under 37 CFR 1.9(c) if that person had made the invention, (2) any concern which would not qualify as a small business concern under 37 CFR 1.9(d) or (3) a nonprofit organization under 37 CFR 1.9(e).

- ☐ no such person, concern, or organization
☐ person, concerns or organizations listed below*

*NOTE: Separate statements are required from each named person, concern or organization having rights to the invention as to their status as small entities. (37 CFR 1.27)

Full Name _____
Address _____
☐ INDIVIDUAL ☐ SMALL BUSINESS CONCERN ☐ NONPROFIT ORGANIZATION

Full Name _____
Address _____
☐ INDIVIDUAL ☐ SMALL BUSINESS CONCERN ☐ NONPROFIT ORGANIZATION

III. ACKNOWLEDGEMENT OF DUTY TO NOTIFY PTO OF STATUS CHANGE

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

IV. DECLARATION

(check the following item, if desired)

NOTE: The following verification statement need not be made in accordance with the rules published on October 10, 1997, 62 Fed. Reg. 52131, effective December 1, 1997.

NOTE: "The presentation to the Office (whether by signing, filing, submitting, or later advocating) of any paper by a party, whether a practitioner or non-practitioner, constitutes a certification under § 10.18(b) of this chapter. Violations of § 10.18(b)(2) of this chapter by a party, whether a practitioner or non-practitioner, may result in the imposition of sanctions under § 10.18(c) of this chapter. Any practitioner violating § 10.18(b) may also be subject to disciplinary action. See §§ 10.18(d) and 10.23(c)(15)." 37 CFR 1.4(d)(2).

- ☐ I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

V. SIGNATURES

(complete only (e) or (f) below)

(e)

NOTE: All inventors must sign the statement.

Name of Inventor

Date: _____

Signature of Inventor

Name of Inventor

Date: _____

Signature of Inventor

Name of Inventor

Date: _____

Signature of Inventor

(add lines for any additional inventors who must sign)

or

(f)

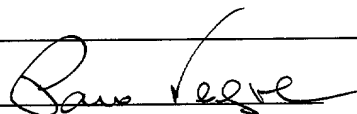
NOTE: The title of the person signing on behalf of a concern or nonprofit organization should be specified.

Name of Person Signing FRANCISCO NEGRE MEDALL

Title of Person Proxy
(if signing on behalf of a concern or non-profit organization)

Address of Person Signing Avda. Hermanos Bou, 27 6(C, 12003
Castellón, Spain

SIGNATURE



DATE 1 Oct. 2001

09/914029

1

PIGMENTS BASED ON SILICA AND IRON OXIDE AND PROCESS FOR THE
MANUFACTURE THEREOF

Field of the Invention

5 The present invention relates to the technical field
of pigment manufacturing. Specifically, it deals with
obtaining silica and iron oxide pigments useful in different
industries and particularly in the ceramics industry. The
silica component of these pigments would be obtained from
10 microsilica or from fumed silica as the most characteristic
feature of the invention.

Description of the state of the art

20 The natural pigments, silica - iron, have been known
since ancient times for their chromophore properties.
15 Extensively used in ceramics for colouring the mass, they
provide a colour within the range of ochre - browns -
oranges - reddish, on being introduced in high ratios which
range from 2 to 12% by weight. However, they essentially
lack homogeneity due to variations in the physico-chemical
20 characteristics of the deposits.

Several efforts have been carried out trying to
reproduce the features of this type of natural colorants,
although these efforts have always come up against the
limitation of achieving good quality products at a
25 competitive cost relative to the market prices established
for naturally occurring products.

In the prior developments there are different attempts
to reproduce this type of colorants based on wet reaction
processes, obtaining a gel, $\text{Fe}_2\text{O}_3 \cdot x\text{SiO}_2$, with a Fe_2O_3 content
30 of 5 to 15% by weight, which must be submitted to a drying,
calcination and milling process in order to obtain the
product with an adequate quality.

In US patent 3,005,724 (1961), the starting product is
a colloidal silica as a silica source, and a source of iron
35 oxide, preferably iron sulphate, obtaining a suspension
which is gelified by the addition of an alkaline solution.
This gel is dried and calcinated at temperatures between 900

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and 1400° C and afterwards it is milled in order to obtain the pigment.

On the other hand, there are patents in which fumed silica is used to improve the features of the iron oxide synthetic pigments. In these cases, low silica ratios between 0.25 and 10 % of SiO₂ by weight are used, and it is used to improve the flowability and the coloration of the pigments in comparison with the synthetic iron oxides.

US patents 4,221,607 and 4,229,635, are disclosing processes for obtaining this type of iron oxide, starting from a solution of copperas as a source of iron oxide, to which a small proportion of silica is added (between 0.25 and 10% by weight) to obtain the iron oxide pigments with improved properties after a drying and calcination process.

In US patent 4,221,607, the addition of silica is carried out before drying and calcination and an iron pigment is obtained which behaves better in the calcination process, while in US patent 4,229,635, the addition of iron is carried out after the calcination process and during a washing process of the colorant, before final drying and milling, obtaining a pigment with improved flowability.

References have not been found to the use of microsilica for the manufacture of silica-iron oxide pigments.

Brief description of the invention

The object of the present invention was to obtain inorganic pigments of the silica-iron oxide, $X \text{ Fe}_2\text{O}_3$ and SiO_2 system, using microsilica or fumed silica as the silica source, along with the process for industrial obtention of said pigment. The obtained pigments may have a of red - orange hue colouration, above all when used in the manufacture of ceramic products of low porosity such as porcelain stoneware. They are a competitive alternative, in terms of quality and cost, to natural materials such as Thiviers stoneware, which are currently used. The presentation of the product is in the form of a micronised powder. The product can be incorporated into the base

Detailed description of the invention

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basic mixtures to improve the features of the colorant and/or to modify its coloration.

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stable during its use in ceramic compositions that are submitted to high temperatures during their manufacture.

These colorants of silica-iron are obtained by calcination of mixtures of raw materials at high temperatures, forming a pigment with tridymite and/or cristobalite structure in which the iron oxide is incorporated with hematite structure. The tridymite or cristobalite are formed by calcination of the amorphous silica, obtaining a stable structure or another one, depending on the firing conditions and on the present additives and impurities.

The different compositions of raw material can be prepared through dry or wet processes. In the dry process, the mixture of raw materials is carried out in a mill or a blender. The resulting mixture may optionally be granulated to facilitate its handling (transport, dosing...). In the wet process, a suspension in water of the raw materials is prepared by milling and dispersion, drying the mixture in an atomisation process, in which an agglomerated material suitable for subsequent processing is obtained.

These mixtures of raw materials, more or less agglomerated, may optionally pass through a prior drying stage, to then proceed to calcination (thermal treatment at high temperature in which the different physico-chemical transformations that convert the material into its finished state will take place). Said calcination may be carried out in different oven types at temperatures comprised between 800 and 1300° C, depending on the features of the silica, the source of iron oxide and the used additives.

In some cases, a prior calcination may be effected, at a lower temperature to suitably prepare the raw materials for their subsequent reaction. In this stage, if necessary, the present organic material and the iron oxide are oxidised and/or the raw material acting as a source of iron oxide is decomposed.

After calcination the product is submitted to a particle size reduction step to provide a more homogeneous

colour. Said operation is preferably carried out in a dry milling installation, with dynamic classification which permits to assure that more than 99 % of the particles are smaller than 40 micrometers.

5 Examples

Example 1

87 kg of microsilica and 13 kg of synthetic red iron oxide are milled in a silex-ball mill for 4 hours with 50 litres of water. The suspension obtained is dried and the granulate obtained calcinated at a maximum temperature of 1050° C for three hours. A red coloured product is obtained, which is milled in a mill with alumina balls in dry conditions, until 99 % of the particles are smaller than 40 microns. This colorant, added to a standard composition of porcelain stoneware in a ratio of 5 % by weight, provides fired pieces with Hunter-LAB chromaticity coordinates of L=38.0 a=15.6 b=7.4.

Example 2

346 kg of microsilica and 72 kg of synthetic red iron oxide are granulated in an intensive granulation machine with 80 litres of water. The granulate obtained is dried and calcinated at 1025° C for 6 hours at this maximum temperature. A reddish coloured product is obtained, which is milled in the same way as example 1. The chromaticity coordinates that are obtained are L=40.3, a=15.0 and b=7.3.

Example 3

94 kg of microsilica and 6 kg of synthetic red iron oxide are processed in the same way as in example 1. The chromaticity coordinates that are obtained are L=43.5, a=17.2 and b=8.5.

Example 4

A suspension is prepared with 65.8 kg of microsilica, 34.2 kg of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and 75.0 kg of water in a silex-ball mill. The milling lasts 4 hours. The suspension is dried and a granulate obtained that is pre-calcinated to decompose the sulphates and to oxidise the iron oxide and then the mixture is calcinated at 1100° C.

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The chromaticity coordinates that are obtained for the fired pieces prepared with 5% by weight of colorant are L.43.0, a=16.6 and b=10.5.

Description of the figures

5 Figure 1. Block diagram of the manufacturing process used.

1. Microsilica
2. Source of iron oxide
3. Milling
- 10 4. Agglomeration
5. Pre-calcination
6. Calcination
7. Cooling
8. Blending
- 15 9. Dry milling
10. Blending
11. Sacking

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CLAIMS

1. Pigments of silica-iron oxide characterised in that the silica component (SiO_2) is obtained from microsilica or fumed silica, having a ratio of silica that ranges between 70 and 98% by weight, while the ratio of iron oxide ranges between 2 and 30% by weight.

2. A process for obtaining pigments of claim 1, characterised in that it comprises the following steps:

- 10 a) Blending the majority and minority raw materials,
- b) Agglomerating of the resulting blend of raw materials from the previous stage
- c) Calcinating in an oven the agglomerated blend obtained in the previous stage with a thermal
15 cycle at temperatures comprised between 800 and 1300° C, with residence times ranging between 1 and 24 h,
- d) Blending the pigment obtained in the previous calcination step,
- 20 e) Reducing the particle size of the obtained blend of pigment,
- f) Final blending with control of the chromaticity coordinates of the obtained pigment,
- g) Dosing and packaging.

25 3. A process according to claim 2, in which stages a) and/or b) may be carried out in dry conditions.

4. A process according to claim 2, in which stages a) and/or b) may be carried out in wet conditions.

30 5. A process according to claims 2 and 3, in which the blend from stage a) is carried out by milling.

6. A process according to claims 2 and 4, in which the mixture from stage a) is carried out by dispersion, preferably in water.

35 7. A process according to claims 2, 3 and 5, in which the agglomeration of stage b) consists of a granulation.

8. A process according to claims 2, 4 and 6, in which the agglomeration of stage b) consists of drying by

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atomisation.

9. A process according to claims 2 to 8, in which optionally, before stage c) there is a prior pre-calcination step.

5 10. A process according to claims 2 to 9, in which optionally, after stage c), there is a cooling step, prior to blending of the resulting pigment.

11. A process according to claims 2 to 10, in which step e) preferably consists of grinding or milling.

10 12. Pigments obtained according to the process of claims 2 to 11.

13. Use of the microsilica as a source of SiO_2 in the manufacture of inorganic pigments and/or colorants.

15 14. Use according to claim 13, characterised in that the source of silica is obtained from condensation of gases evolved during the manufacture of silicon metal and/or alloys thereof.

20 15. Use of the pigments of claims 1 or 12, alone or in blends with other materials, as integrants in the compositions of enamels, glasses, ceramics, cements, plastics, laminates, graphic inks and rubber.

25 16. Use of the pigments of claims 1 or 12, alone or in blends with other materials, in the surface decoration of enamels, glasses, ceramics, cements, plastics, laminates, graphic inks and rubber.

17. A ceramic product characterised in that it includes in its composition the pigments of claims 1 or 12.

18. A ceramic product in accordance with claim 17, characterised in that it consists of a porcelain stoneware.

30 19. A porcelain stoneware in accordance with claim 18, characterised in that it shows chromatic coordinates (Hunter-LAB) in the following ranges: $L = 36-46$, $a = 10-18$ and $b = 7-11$, for a percentage pigment of 2% that gives a colour of red-orange tone.

ABSTRACT

Pigments based on silica and iron oxide and manufacturing process thereof. In order to obtain a pigment
5 of silica-iron oxide, microsilica is blended and/or milled
with a source of iron oxide in ratios between 2 and 30 % by
weight of iron oxide. With the blending of raw materials a
dry agglomerate is obtained that is calcinated at
10 temperatures between 800 and 1300° C, obtaining a product
that is milled to obtain a powder of red colour. This
colorant can be used in a wide range of uses, for example,
added to a standard porcelain stoneware composition, it
allows red and orange coloured pieces to be obtained.

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Optional Customer No. Bar Code



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PATENT TRADEMARK OFFICE

COMBINED DECLARATION AND POWER OF ATTORNEY

(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT, SUPPLEMENTAL, DIVISIONAL,
CONTINUATION, OR C-I-P)

As a below named inventor, I hereby declare that:

TYPE OF DECLARATION

This declaration is of the following type:

(check one applicable item below)

- ☐ original.
☐ design.

NOTE: With the exception of a supplemental oath or declaration submitted in a reissue, a supplemental oath or declaration is not treated as an amendment under 37 CFR 1.312 (Amendments after allowance). M.P.E.P. Section 714.16, 7th Ed.

- ☐ supplemental.

NOTE: If the declaration is for an International Application being filed as a divisional, continuation or continuation-in-part application, do not check next item; check appropriate one of last three items.

- ☒ national stage of PCT.

NOTE: If one of the following 3 items apply, then complete and also attach ADDED PAGES FOR DIVISIONAL, CONTINUATION OR C-I-P.

NOTE: See 37 C.F.R. Section 1.63(d) (continued prosecution application) for use of a prior nonprovisional application declaration in the continuation or divisional application being filed on behalf of the same or fewer of the inventors named in the prior application.

- ☐ divisional.
☐ continuation.

NOTE: Where an application discloses and claims subject matter not disclosed in the prior application, or a continuation or divisional application names an inventor not named in the prior application, a continuation-in-part application must be filed under 37 C.F.R. Section 1.53(b) (application filing requirements-nonprovisional application).

- ☐ continuation-in-part (C-I-P).

INVENTORSHIP IDENTIFICATION

WARNING: *If the inventors are each not the inventors of all the claims, an explanation of the facts, including the ownership of all the claims at the time the last claimed invention was made, should be submitted.*

My residence, post office address and citizenship are as stated below, next to my name. I believe that I am the original, first and sole inventor (*if only one name is listed below*) or an original, first and joint inventor (*if plural names are listed below*) of the subject matter that is claimed, and for which a patent is sought on the invention entitled:

TITLE OF INVENTION

Pigments Based on Silica and Iron Oxide and Process for the Manufacture Thereof

SPECIFICATION IDENTIFICATION

The specification of which:

(complete (a), (b), or (c))

(a) ☐ is attached hereto.

NOTE: *"The following combinations of information supplied in an oath or declaration filed on the application filing date with a specification are acceptable as minimums for identifying a specification and compliance with any one of the items below will be accepted as complying with the identification requirement of 37 C.F.R. Section 1.63:*

"(1) name of inventor(s), and reference to an attached specification which is both attached to the oath or declaration at the time of execution and submitted with the oath or declaration on filing;

"(2) name of inventor(s), and attorney docket number which was on the specification as filed; or

"(3) name of inventor(s), and title which was on the specification as filed."

Notice of July 13, 1995 (1177 O.G. 60).

(b) ☐ was filed on _____, ☐ as Application No. _____
☐ and was amended on _____ (if applicable).

NOTE: *Amendments filed after the original papers are deposited with the PTO that contain new matter are not accorded a filing date by being referred to in the declaration. Accordingly, the amendments involved are those filed with the application papers or, in the case of a supplemental declaration, are those amendments claiming matter not encompassed in the original statement of invention or claims. See 37 C.F.R. Section 1.67.*

NOTE: *"The following combinations of information supplied in an oath or declaration filed after the filing date are acceptable as minimums for identifying a specification and compliance with any one of the items below will be accepted as complying with the identification requirement of 37 C.F.R. Section 1.63:*

(A) *application number (consisting of the series code and the serial number, e.g., 08/123,456);*

(B) *serial number and filing date;*

(C) *attorney docket number which was on the specification as filed;*

(D) *title which was on the specification as filed and reference to an attached specification which is both attached to the oath or declaration at the time of execution and submitted with the oath or declaration; or*

(E) *title which was on the specification as filed and accompanied by a cover letter accurately identifying the application for which it was intended by either the application number (consisting of the series code and the serial number, e.g., 08/123,456), or serial number and filing date. Absent any statement(s) to the contrary, it will be presumed that the application filed in the PTO is the application which the inventor(s) executed by signing the oath or declaration.*

M.P.E.P. Section 601.01(a), 7th ed.

- (c) ☒ was described and claimed in PCT International Application No. ES99/00060 filed on March 10, 1999 and as amended under PCT Article 19 on _____ (if any).

SUPPLEMENTAL DECLARATION (37 C.F.R. Section 1.67(b))

(complete the following where a supplemental declaration is being submitted)

- ☐ I hereby declare that the subject matter of the
- ☐ attached amendment
- ☐ amendment filed on _____.

was part of my/our invention and was invented before the filing date of the original application, above identified, for such invention.

ACKNOWLEDGMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information, which is material to patentability as defined in 37, Code of Federal Regulations, Section 1.56,

(also check the following items, if desired)

- ☐ and which is material to the examination of this application, namely, information where there is a substantial likelihood that a reasonable Examiner would consider it important in deciding whether to allow the application to issue as a patent, and
- ☐ in compliance with this duty, there is attached an information disclosure statement, in accordance with 37 C.F.R. Section 1.98.

PRIORITY CLAIM (35 U.S.C. Section 119(a)-(d))

NOTE: "The claim to priority need be in no special form and may be made by the attorney or agent if the foreign application is referred to in the oath or declaration as required by Section 1.63. The claim for priority and the certified copy of the foreign application specified in 35 U.S.C. Section 119(b) must be filed in the case of an interference (Section 1.630), when necessary to overcome the date of a reference relied upon by the examiner, when specifically required by the examiner, and in all other situations, before the patent is granted. If the claim for priority or the certified copy of the foreign application is filed after the date the issue fee is paid, it must be accompanied by a petition requesting entry and by the fee set forth in Section 1.17(i). If the certified copy is not in the English language, a translation need not be filed except in the case of interference; or when necessary to overcome the date of a reference relied upon by the examiner; or when specifically required by the examiner, in which event an English language translation must be filed together with a statement that the translation of the certified copy is accurate." 37 C.F.R. Section 1.55(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

(complete (d) or (e))

- (d) ☒ no such applications have been filed.
(e) ☐ such applications have been filed as follows.

NOTE: Where item (c) is entered above and the International Application which designated the U.S. itself claimed priority check item (e), enter the details below and make the priority claim.

**PRIOR FOREIGN/PCT APPLICATION(S) FILED WITHIN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS APPLICATION
AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. SECTION 119(a)-(d)**

COUNTRY (OR INDICATE IF PCT)	APPLICATION NUMBER	DATE OF FILING DAY, MONTH, YEAR	PRIORITY CLAIMED UNDER 35 USC 119
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO
			<input type="checkbox"/> YES <input type="checkbox"/> NO

**CLAIM FOR BENEFIT OF PRIOR U.S. PROVISIONAL APPLICATION(S)
(35 U.S.C. Section 119(e))**

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below:

PROVISIONAL APPLICATION NUMBER

_____/_____
_____/_____
_____/_____

FILING DATE

**CLAIM FOR BENEFIT OF EARLIER U.S./PCT APPLICATION(S)
UNDER 35 U.S.C. SECTION 120**

- ☐ The claim for the benefit of any such applications are set forth in the attached
ADDED PAGES TO COMBINED DECLARATION AND POWER OF ATTORNEY
FOR DIVISIONAL, CONTINUATION OR CONTINUATION-IN-PART (C-I-P)
APPLICATION.

ALL FOREIGN APPLICATION(S), IF ANY, FILED MORE THAN 12 MONTHS
(6 MONTHS FOR DESIGN) PRIOR TO THIS U.S. APPLICATION

NOTE: If the application filed more than 12 months from the filing date of this application is a PCT filing forming the basis for this application entering the United States as (1) the national stage, or (2) a continuation, divisional, or continuation-in-part, then also complete ADDED PAGES TO COMBINED DECLARATION AND POWER OF ATTORNEY FOR DIVISIONAL, CONTINUATION OR C-I-P APPLICATION for benefit of the prior U.S. or PCT application(s) under 35 U.S.C. Section 120.

POWER OF ATTORNEY

I hereby appoint the following practitioner(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

(list name and registration number)

JOSEPH H. HANDELMAN, 26179

JULIAN H. COHEN, 20302

JOHN RICHARDS, 31053

WILLIAM R. EVANS 25858

RICHARD J. STREIT, 25765

JANET I. CORD, 33778

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CYNTHIA R. MILLER, 34678

RICHARD P. BERG, 28145

(Check the following item, if applicable)

- ☐ I hereby appoint the practitioner(s) associated with the Customer Number provided below to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.
- ☐ Attached, as part of this declaration and power of attorney, is the authorization of the above-named practitioner(s) to accept and follow instructions from my representative(s).

NOTE: "Special care should be taken in continuation or divisional applications to ensure that any change of correspondence address in a prior application is reflected in the continuation or divisional application. For example, where a copy of the oath or declaration from the prior application is submitted for a continuation or divisional application filed under 37 CFR 1.53(b) and the copy of the oath or declaration from the prior application designates an old correspondence address, the Office may not recognize, in the continuation or divisional application, the change of correspondence address made during the prosecution of the prior application. Applicant is required to identify the change of correspondence address in the continuation or divisional application to ensure that communications from the Office are mailed to the current correspondence address. 37 CFR 1.63(d)(4)." Section 601.03, M.P.E.P., 7th Ed

SIGNATURE(S)

NOTE: Carefully indicate the family (or last) name, as it should appear on the filing receipt and all other document.

NOTE: Each inventor must be identified by full name, including the family name, and at least one given name without abbreviation together with any other given name or initial, and by his/her residence, post office address and country of citizenship. 37 C.F.R. Section 1.63(a)(3).

NOTE: Inventors may execute separate declarations/oaths provided each declaration/oath sets forth all the inventors. Section 1.63(a)(3) requires that a declaration/oath, inter alia, identify each inventor and prohibits the execution of separate declarations/oaths which each sets forth only the name of the executing inventor. 62 Fed. Reg. 53,131, 53,142, October 10, 1997,

Full name of sole or first inventor

VICENTE BAGAN VARGAS
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(check proper box(es) for any of the following added page(s))

**ADDED PAGE TO COMBINED DECLARATION AND POWER OF
ATTORNEY FOR SIGNATURE BY FOURTH AND SUBSEQUENT INVENTORS**

Full name of fourth joint inventor, if any

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Inventor's signature (X) Vicente Beltran Porcar

Date (X) 29-11-2001 Country of Citizenship SPAIN

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Inventor's signature (X) Benito Cervantes Rodriguez

Date (X) 30/11/01 Country of Citizenship SPAIN

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Full name of sixth joint inventor, if any

(Given Name) (Middle Initial or Name) Family (Or Last Name)

Inventor's signature

Date Country of Citizenship

Residence

Post Office Address

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New York, N.Y. 10023

DIRECT TELEPHONE CALLS TO:
(Name and telephone number)

CLIFFORD J. MASS
(212) 708-1890

(complete the following if applicable)

Since this filing is a [] continuation [] divisional there is attached hereto a Change of Correspondence Address so that there will be no question as to where the PTO should direct all correspondence.

DECLARATION

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

that form a part of this declaration)

[X] **Signature** for fourth and subsequent joint inventors. *Number of pages added* 1

* * *

[] **Signature** by administrator(trix), executor(trix) or legal representative for deceased or incapacitated inventor. *Number of pages added* _____

* * *

[] **Signature** for inventor who refuses to sign or cannot be reached by person authorized under 37 C.F.R. Section 1.47. *Number of pages added* _____

* * *

[] Added page for **signature** by one joint inventor on behalf of deceased inventor(s) where legal representative cannot be appointed in time. (37 C.F.R. Section 1.47)

* * *

[] Added pages to combined declaration and power of attorney for divisional, continuation, or continuation-in-part (C-I-P) application.

[] Number of pages added

* * *

[] Authorization of practitioner(s) to accept and follow instructions from representative.

*(If no further pages form a part of this Declaration,
then end this Declaration with this page and check the following item)*

[] This declaration ends with this page.